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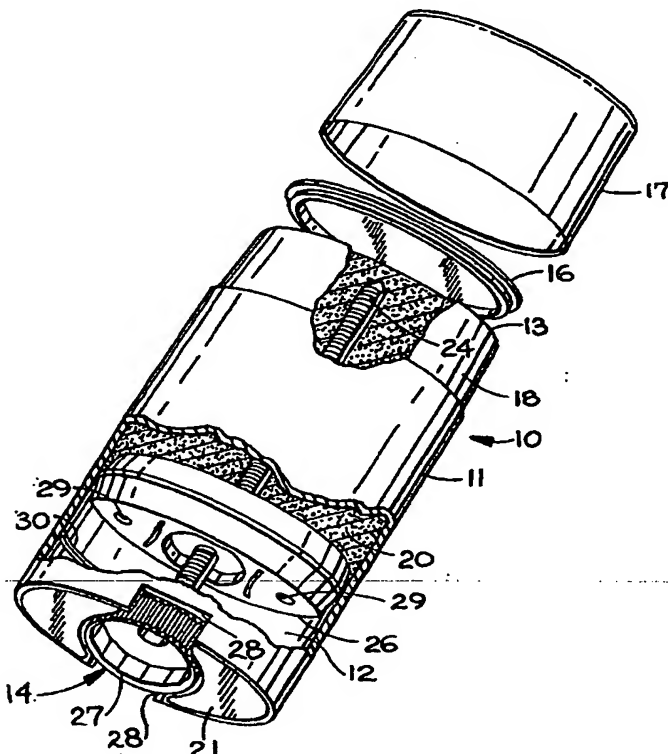
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<b>(21) International Application Number:</b> PCT/US94/13903 <b>(22) International Filing Date:</b> 6 December 1994 (06.12.94)  <b>(30) Priority Data:</b> 08/176,535 30 December 1993 (30.12.93) US  <b>(71) Applicant:</b> MINNESOTA MINING AND MANUFACTURING COMPANY [US/US]; 3M Center, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).  <b>(72) Inventors:</b> FITCH, Joanne; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). KUMAR, Kanta; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). PINAULT, Duane, M.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US).  <b>(74) Agents:</b> BUSSE, Paul, W. et al.; Minnesota Mining and Manufacturing Company, Office of Intellectual Property Counsel, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).		<b>(81) Designated States:</b> AU, BR, CA, JP, NZ, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>

**(54) Title:** ECTOPARASITE CONTROL STICK FOR DOMESTICATED ANIMALS

**(57) Abstract**

This invention provides an ectoparasite control composition for topical application to the hair coat or fur of a domesticated animal or household pet, such as a dog or cat, to control ectoparasites, such as fleas or ticks. In another aspect, the invention provides an applicator package for applying an ectoparasite control composition, in the form of a molded stick, by rubbing or spreading it on the hair coat of such animal and thereby coating it with the composition. In further aspects, the invention provides a method of making ectoparasite control compositions and to a method of applying the same.



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## ECTOPARASITE CONTROL STICK FOR DOMESTICATED ANIMALS

5           This invention provides an ectoparasite control composition for topical application to the hair coat or fur of a domesticated animal or household pet, such as a dog or cat, to control ectoparasites, such as fleas or ticks. In another aspect, the invention provides an applicator package for applying an ectoparasite control composition, in the form of a molded stick, by rubbing or spreading it on the hair  
10 coat of such animal and thereby coating it with the composition. In further aspects, the invention provides a method of making ectoparasite control compositions and to a method of applying the same.

Man is constantly at war with harmful pests that annoy, bite, and cause infections. Various compositions and application techniques are known for  
15 controlling or eliminating biting or blood-sucking pests (ectoparasites), such as fleas, ticks, flies, and lice, which often irritate or infest animals. Of particular concern is the presence and effect of such pests on household pets or companion animals, such as dogs and cats, and other domesticated animals, such as horses. "Fleas are big trouble-and big business," according to The Wall Street Journal,  
20 December 28, 1993, page A1, and Americans spend one billion dollars a year on various products to kill a species of fleas that preys on both dogs and cats.

Over the years, the patent literature has reported a host of aerosols and space sprays, liquids, soaps, shampoos, wettable powders, granules, baits, and dusts, as well as various devices or articles, such as insecticidal ear tags, tail tags,  
25 collars, strips, and applicators that contain push-up insecticide stick formulations, for the control of pests.

A recent composition for control of insects is the insect repellent/toxicant composition described in Canadian Patent 1,286,985 (Matteson et al.). This composition is substantive to animal hide and hair and contains a pyrethroid  
30 insecticide, a certain water-insoluble acrylate polymer, and a liquid carrier.

A number of patents describe insect repellents or insecticides in the form of a stick. U.S. Patent 3,162,575 (Lang) describes controlling face flies on livestock by applying a composition, preferably in the form of a stick, that includes an insecticide, microcrystalline wax, and an oil to an animal's face.  
35 U.S. Patent 3,826,232 (Duffey et al.) describes a pest control stick formulation that may be applied to the neck or face of domesticated animals to eliminate and control pests, particularly fleas, on dogs and cats. The stick is dispensed from a

tubular receptacle and contains a certain carbamate as the active ingredient and, as a bodying or carrying agent, a polyethylene glycol or derivative. The stick formulation also includes fatty acid such as stearic acid, and fatty alcohol, such as cetyl alcohol.

- 5           Insect repellent sticks which liquify when rubbed on the human skin are described in U.S. Patent 2,819,995 (Wassell). These sticks have components that are insoluble in water and contain an active ingredient, a fatty acid such as stearic acid, an ozokerite wax and no soap (the stick being said to overcome the disadvantages of a soap-gel repellent stick containing an active ingredient and  
10       sodium stearate). An insect repellent alcoholic soap gel stick for application to the skin area is described in U.S. Patent 2,465,470 (Omohundro et al.).

          U.S. Patent 4,473,582 (Greene) describes an insecticidal package for use against crawling insects. This package includes a cylindrical tube-like receptacle containing a solid insecticidal stick composition of an insecticide, a fatty  
15       hydrocarbon monoether of propylene glycol, monoethanolamide of fatty acid, and, optionally, a lubricating agent, such as silicone oil (polysiloxane) to facilitate movement of the insecticidal stick within the receptacle. The stick is rubbed onto the surface to be treated, usually along areas where the target insects have been seen or where they may find harborage, e.g., a baseboard.

- 20           Insecticidal livestock ear tags, tail tags, pet collars, or strips, comprising an insecticide dispersed in a thermoset matrix, are described in recent U.S. Patent 5,194,265 (Boettcher et al.).

          A gellable slow release insecticidal composition for various animals is described in U.S. Patent 4,762,718 (Marks). It contains a film-former, such as  
25       hydroxypropyl cellulose, and an in situ insecticide.

- The present invention, in one aspect, provides a solid ectoparasite control composition, which can be in the form of a shaped article, such as a stick, that is an elongated shape, preferably cylindrical or other applicable form, which is adapted to be rubbed or spread by hand, for example, from a push-up receptacle  
30       or applicator tube, on the hair coat or fur of a domesticated animal or household pet, such as a dog, cat, or horse, to transfer or deposit a thin, solid, cosmetically acceptable, substantive coating or layer of the ectoparasite control composition on the hairs of the coat of the animal, for example, its ears, back, or hindquarters, to control, for example to kill or repel, ectoparasites, such as fleas and ticks,  
35       which are on or come on the animal and are likely to cause it to be stressed, uncomfortable, or infected with disease.

The ectoparasite control composition of this invention may be made from a heated precursor liquid mixture or dispersion of components or ingredients which gells or solidifies on cooling into a desired stick or other applicable form, which is soft and abradable, but firm or hard enough to retain its shape during normal storage and use, and to permit an effective topical application to be rubbed on the hair coat of the animal. The components of the composition are stably and homogenously dispersed therein and comprise an ectoparasite control agent or anti-ectoparasite agent, e.g., a pyrethrum insecticide, a water-insoluble acrylate polymer, such as polymer with polymeric acrylate segment(s) with or without polysiloxane segment(s), which imparts substantivity to the applied coating of the ectoparasite control composition and thereby prolongs or extends its ectoparasite control residual activity, e.g., at least 6 days, preferably for 2 weeks or more, and, as a further essential component, a normally solid, inert, water-soluble gelling (or bodying) agent, such as sodium stearate, which gels or solidifies the precursor liquid mixture of the components and sustains the stick shape or other applicable form during normal storage and use thereof e.g., at temperatures up to at least about 110°F (43°C), for example, after it is poured into a dispensing receptacle (such as dispensers used for antiperspirant or deodorant sticks) and cooled to room temperature (20°C).

The stick or other applicable shape may be applied to the hair coat of domesticated animals by rubbing or spreading it on the coat, preferably in the direction of the lay of its hairs, to deposit (for example, on the ears, back, or hindquarters of the animal) a relatively uniformly thin, non-migratory, non-smearing, solid layer or coating of the ectoparasite control composition (without it hardening or liquefying). Magnification has shown the so-treated hairs to be discretely and uniformly coated. The applied coating is in a cosmetically acceptable form, that is, the coating has an appearance and tactibility that is not objectionable to the pet owner or animal.

The applied coating is generally transparent or translucent and may be colorless, light yellow or other aesthetically and cosmetically acceptable color. The applied coating is typically somewhat shiny and cohesive but not tacky or sticky or messy, it will feel smooth (not crumbly or friable or stiff) and somewhat oily or slippery or lubricious, and the treated coat is not matted when the ectoparasite control composition is applied to deposit only the relatively uniformly thin coating necessary to provide the desired ectoparasite control. The applied coating is

mycologically stable and not toxic or irritating to mucous membrane and has no objectionable odor. The coating is normally solid at the normal body and skin temperatures of the animal.

The effective activity of the ectoparasite control agent is long due to the substantivity imparted by the polymer component, and the ectoparasite control agent does not "bleed" or "bloom" from the stick or coating thereof. As such, the normal activity of the animal, for example, its running or laying down on the ground, carpet, upholstery, etc., and the normal exposure to the elements, such as rain, will not significantly remove the applied coating or significantly lessen the effective activity of the active control ingredient, that activity lasting, for example, 6 to 14 days or more, during which time the cosmetic acceptability of the coating also is not significantly lessened. The amount of ectoparasite control composition applied will be sufficient for effective activity and generally will be 0.1 to 0.3 grams/kilogram of animal body weight. If needed, the application of the control composition to the animal may be repeated. If desired, the coating, though water resistant or repellent, can be removed from the hair coat by washing it with warm water and a mild detergent or shampoo, such as is normally used to maintain cleanliness of the animal.

In the accompanying drawing, FIG. 1 is an isometric view of an embodiment of the ectoparasite control package in the form of a push-up type applicator for dispensing an ectoparasite control stick of this invention which is also shown in the isometric view of FIG. 2. FIG. 3 is a view in elevation and detail of the rotatable means shown in FIG. 1.

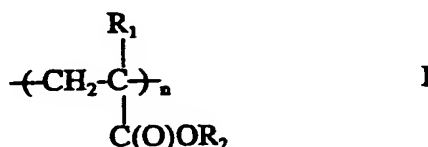
The ectoparasite control agents which can be used as the active components or ingredients of the compositions of this invention are generally substances (or group of substances, such as those specified by the U.S. Environmental Protection Agency) that will prevent, destroy, repel or mitigate any ectoparasites that commonly are on or infest domesticated animals, particularly household pets or companion animals, for example, dogs and cats. Such ectoparasites are typically fleas, e.g., *C. felis*, and ticks, e.g., *R. sanguineus* and *D. variabilis*. Such ectoparasite control agents, or anti-ectoparasitic agents, may be an insecticide, pheromone, insect growth regulator, or repellent, for example, those described and listed in said U.S. Patent 5,194,265 (Boettcher et al.). Generally, the classes of insecticides which can be used include known organophosphates such as chlorpyrifos, synthetic and natural pyrethrins such as permethrin, and carbamates such as propoxur.

The ectoparasite control agents used in this invention may be in the form of dilute solutions in organic solvents, which may also include stabilizers and inert materials, and may be used together with known synergists which enhance the activity of the agents. A particularly useful agent is a contact pyrethrum insecticide, such as that product sold as "Kenya Pyrethrum Extract Refined Concentrate," E.P.A. Registration No. 4713-5, a light amber liquid which, according to its Material Safety Data Sheet (November 7, 1989), contains 54% pyrethrins, 16% isoparaffinic solvent, 5% (max.) 2,6-di-tert-butyl-4-methylphenol or "butylated hydroxytoluene" (BHT) stabilizer, and 25% inert materials. Synergists which may be used with pyrethrum and pyrethroid insecticides include piperonyl butoxide and others described on p. 195-197 of Chap. 10 of "Pyrethrum the Natural Insecticide," edited by J.E. Casida, Academic Press, New York (1973).

Stabilizers, that is, antioxidants and ultraviolet absorbers, which may be used with the active anti-ectoparasite agent include those described in Canadian Patent 1,286,985.

The organic solvents which may be used to prepare a solution of the active ectoparasite control agent, e.g., as a 20 to 60 wt% solution, as a component in the compositions or sticks of this invention, include those normally used in supplying commercial products of such agents, such as isoparaffins, kerosene, ethanol, acetone and ethylene dichloride.

The polymer component of the ectoparasite control composition which imparts substantivity to the coating applied to the animal so as to prolong the residence of the active ingredient on the hair coat of the animal, is generally an acrylate polymer. One class of such a polymer may be represented by the general formula:



wherein:

n is an integer which represents the number of repeating units shown in parenthesis, n generally being 10 to 100,000, preferably 50 to 5000;

each R<sub>1</sub> in a repeating unit is independently a hydrogen atom or a methyl group, -CH<sub>3</sub>;

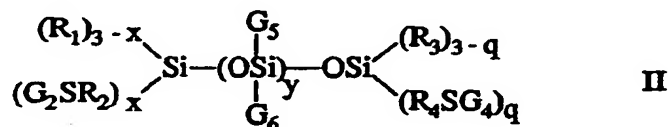
each  $R_2$  in a repeating unit is independently a hydrogen atom, a hydrocarbon moiety (such as an alkyl group, with 1 to 20 carbon atoms, e.g., isobutyl, or a cycloalkyl group with 3 to 8 carbon atoms, e.g., cyclohexyl), an organic moiety containing a fluoroaliphatic group, for example, derived from the

5 "A" monomers described in U.S. Pat. No. 4,972,037 (such as a N-alkyl-perfluoroalkylsulfonamidoalkylene, e.g.,  $-\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)\text{SO}_2\text{C}_8\text{F}_{17}$ ), or a poly(dialkylsiloxane) group (e.g.,  $-(\text{CH}_2)_3[\text{Si}(\text{CH}_3)_2\text{O}]_m\text{Si}(\text{CH}_3)_2(\text{CH}_2)_3\text{CH}_3$ , wherein  $m$  is an integer which represents the number of repeating units shown in brackets,  $m$  generally being 6 to 700, preferably 60 to 300).

10 Another class of acrylate polymer which may be used as the substantive polymer component in the present composition is a vinyl-silicone graft or block copolymer comprising a silicone polymer segment and a vinyl polymeric segment. Such polymer may be made by a polymerization process using a mercapto-functional silicone transfer agent. The weight ratio of vinyl polymer

15 block (segment A in formula II) to silicone segment of the copolymer varies. Preferred copolymers have a weight ratio of vinyl polymer segment to silicone segment ranging from about 98:2 to 40:60, in order that the copolymer possess properties inherent to each of the different polymeric segments. Such copolymer may be represented by the general formula:

20



25

wherein

$R_1$  are monovalent moities which may independently be the same or different and are selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, hydroxyl, hydrogen, and fluoroalkyl;

30

$R_2$  are divalent linking groups which may independently be the same or different;

$R_3$  are monovalent moities which may independently be the same or different and are selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, hydroxyl, hydrogen, and fluoroalkyl;

35

$R_4$  are divalent linking groups which may independently be the same or different;

$x$  is an integer of 0-3;



y is an integer of 10 or greater;

q is an integer of at least 1;

G<sub>5</sub> are monovalent moieties which may independently be the same or different selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, fluoroalkyl, hydrogen, and -ZSA;

A is a vinyl polymeric segment consisting essentially of polymerized free radically polymerizable monomer;

G<sub>6</sub> are monovalent moieties which may independently be the same or different selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, fluoroalkyl, hydrogen, and -ZSA;

G<sub>2</sub> comprises A;

G<sub>4</sub> comprises A; and

Z is a divalent linking group; useful divalent linking groups include (but are not limited to) C<sub>1</sub> to C<sub>10</sub> alkylene, alkarylene, arylene and alkoxyalkylene.

The above-described acrylate polymers include those reported in, for example, U.S. Patents 4,172,122 (Kubik et al.), 4,972,037 (Garbe et al.), 4,981,903 (Garbe), 5,032,460 (Kantner et al.), 5,194,265 (Boettcher) and Canadian Patent 1,286,985 (Matteson et al.). Some examples of such polymers are poly(isobutyl methacrylate-co-N-methyl perfluorooctylsulfonamidoethylacrylate)-g-poly(dimethylsiloxane) and poly(dimethylsiloxane)-g-poly(isobutyl methacrylate), (products SA 70-5 IBMMF and VS 70 IBM listed in commercial bulletin 70-0705-2562-4, 3M, St. Paul, MN); and poly(isooctyl acrylate-co-stearyl methacrylate-co-acrylic acid). These polymers may be used, in the formulation of the compositions or sticks of this invention, as solutions in the solvents used in their polymerization preparation or in other solvents such as aliphatic, aromatic, or oxygen-containing polar solvents, e.g., alcohols, ketones, esters, glycols, silicones, and ethers. Examples of such solvents are propylene glycol and fatty alkyl esters of benzoic acid. Mixtures of these solvents may be used and the solvents may also be used to solubilize the ectoparasite control agent and associated synergist.

The gelling or bodying components typically provide ectoparasite control compositions having melting points greater than 110°F (43°C). Such gelling or bodying components have low or no odor, and are preferably those which are soluble or dispersible in water and the hot precursor liquid, and which may also be incorporated in the compositions as a diluent. Substances which may be used

to gel the precursor liquid and to sustain the shaped forms or sticks include sodium stearate, the preferred gelling agent, and various other compatible gelling agents that readily dissolve in and mix with the precursor liquid mixture.

To facilitate the homogeneity of the water component with the oil or organic components, (ectoparasite control agents, the substantive polymer, and the organic solvents used to formulate the ectoparasite control composition) suitable surfactants may be used, such as those non-ionic surfactants that aid in preparing the precursor liquid mixture as a water-in-oil emulsion. Alkoxylated fatty alcohols, such as polyoxypropylene polyoxyethylene cetyl ether, are particularly useful for emulsifying water into the oily components of the ectoparasite control composition. Generally, the amount of surfactant to be used will be 15 to 45 wt%, preferably 18 to 35, wt%, and the amount of water emulsified will be 15 to 30 wt%.

Other optional components which may be incorporated in the precursor liquid mixture typically enhance application (rubbing or spreading) of the stick and/or the cosmetic acceptability of the applied coating. Lubricity agents for imparting lubricity to the applied coating, such as silicone fluids, are such optional components.

The relative amounts of the various components of the ectoparasite control compositions of this invention vary and the particular amount of each will be that amount which is sufficient, or best, for it to perform its function. The amount of ectoparasite control agent to be used is an amount which provides the desired killing, prevention, repelling, or mitigation of the target ectoparasite over the desired duration. The amount of the polymer component to be used in the ectoparasite control composition is an that amount generally sufficient to impart the desired substantivity to the applied coating, that is, the desired period of effective ectoparasite control, e.g., 14 or more days after application of the stick to the hair coat of the animal to be treated. The amount of gelling agent to be used is an amount sufficient to solidify the precursor liquid mixture upon molding and cooling it to form the desired shape or stick and to sustain it in that form during storage and application to the animal. Table 1 sets forth ranges of amounts of essential and optional components used to prepare ectoparasite control compositions according to this invention.

Table 1

	Component*	Amount, wt%
	Ectoparasite control agent	0.05 to 20, preferably 0.5 to 5
	Synergist	0 to 16
5	Substantive acrylate polymer	0.05 to 5
	Lubricity agent	0 to 10
	Organic solvent	15 to 45
	Water	15 to 30
	Surfactant	15 to 45
10	Gelling agent	2 to 10

- \* Each of the listed components can actually be mixtures of several like components, e.g., the listed "Organic solvent" can represent the solvent in which the ectoparasite control agent is dissolved and formulated and the solvent in which the substantive polymer is dissolved and formulated.

In preferred embodiments of this invention, the ectoparasite control agent is pyrethrin insecticide, the substantive polymer is poly(isobutyl methacrylate-co-N-methyl-perfluorooctylsulfonamidoethyl acrylate) -g-poly(dimethylsiloxane) and the gelling agent is sodium stearate. Table 2 also sets forth ranges of amounts of essential and optional components which may be used to prepare ectoparasite control compositions.

Table 2

	Component	Amount, wt%
	Pyrethrin insecticide, 54 wt% solution in kerosene	0.05 to 4, preferably 0.5 to 3
5	Piperonyl butoxide synergist	0 to 15, preferably 2.2 to 13
10	Poly(isobutyl methacrylate-co-N-methyl-perfluorooctylsulfonamido-ethyl acrylate) -g- poly(dimethylsiloxane) substantive polymer, 25 wt% solution in Finsolv TN	0.05 to 20, preferably 0.5 to 8
	Poly(dimethylsiloxane)cyclic tetramer lubricity agent	0 to 5, preferably 2 to 5
15	C <sub>12</sub> -C <sub>15</sub> alkyl benzoate solvent for substantive polymer	5 to 10, preferably 7 to 10
	Propylene glycol	20 to 30, preferably 22 to 26
	Polyoxypropylene (5) Polyoxyethylene (20) Cetyl ether (surfactant)	15 to 50, preferably 20 to 25
	Water	15 to 30 preferably 18 to 25
20	Sodium stearate	2 to 10 preferably 3 to 6

The ectoparasite control compositions of this invention are typically made by heating for example, to 50 to 90°C, preferably about 70°C, while stirring a precursor mixture of the solvent solution of the substantive polymer, surfactant, and water, to form a water-in-oil emulsion or dispersion, then, while the latter is still hot, adding the gelling agent (in a particulate form, e.g., powder) while stirring to dissolve it, cooling the resulting liquid mixture for example, to 50 to 70°C, preferably to about 60°C, then, while stirring the cooled mixture, adding the ectoparasite control agent (typically as a concentrate in a solvent) and, if used, the synergist and the silicone fluid components. The resulting mixture is then poured into suitable stick molds and allowed to cool to ambient room temperature (e.g., 20°C), during which cooling the composition will solidify in the desired shape or form. (Alternatively, the precursor liquid mixture may be cooled to solidify it in other solid forms, e.g., pellets or bars, which are stored and shipped and later reheated to form a liquid that is poured into a mold and cooled to form the stick.)

Push-up type applicators or packages may be used to mold the sticks and dispense the coating to the hair coat of the animal. Push-up applicators of this type are described, for example, in U.S. Patents 2,818,167 (McKinley), 3,826,232 (Duffey), and 4,473,582 (Greene).

5        FIG. 1 illustrates an embodiment of package 10 which is used to provide ectoparasite control compositions of this invention. Package 10 comprises a tubular or cylindrical body or casing 11, which is preferably made of molded thermoplastic, such as polyethylene, having an oval cross-section and an internal chamber 12 defined by the inner surface or wall of the tubular body. The tubular  
10 body 11 has an open end 13 and a removable member 14 press-fitted in the other open end. The open end 13 can be closed by press-fitting into it a closure member 16. A removable cap 17, likewise oval in cross-section, is adapted to be press-fitted over a tapered portion 18 of the tubular body 11 to enclose the open end 13 with (or without) the member 16 in place. Disposed within the chamber 12 is the  
15 ectoparasite control stick 20, which is likewise cylindrical and oval in cross-section but can be slightly smaller in that respect so that it can be slid easily in and out of the chamber, the length of the stick, as initially molded, being somewhat shorter than the length of the chamber.

The removable member 14, shown in detail in FIG. 3, comprises a cap 21,  
20 which likewise is oval in cross-section and press-fitted within the end of the chamber 12, and it has a suitable journal 22, in which a rotatable, threaded stem 24 is axially mounted and adapted to axially pass through a piston 26 which is likewise oval in cross-section and adapted to slide back and forth within chamber 12 of the tubular body 11. The stem 24 is affixed on one end to a knob 27 whose  
25 outer surface can be knurled as shown and rotated by hand, the tubular body 11 having opposite-disposed cut-out portions 28 to provide access to the knob for the purpose of such hand operation, which can push the stick 20 in or out of chamber 12 when closure member 16 and cap 17 have been removed.

To expose the stick 20 for application to the hair coat of the animal, cap  
30 17 and closure member 16 are removed and knob 27 is rotated in a clock-wise manner to push the stick out the open end 13 sufficiently to expose the adjacent free end of the stick so that, by then grasping the tubular body 11 by hand, the exposed end of the stick can be rubbed on the hair coat for purposes of applying a coating of the ectoparasite control composition. After application of the coating,  
35 knob 27 can be rotated in the opposite manner to retract stick 20 back into chamber 12, and closure member 16 and cap 17 can be replaced to protect the stick from damage.

In preparing package 10, the stick precursor liquid mixture can be poured into chamber 12 of the empty tubular body 11 when it is in a vertical position with its end closure member 16 (and cap 17, if desired) in place, and with the member 14 and piston 26 removed, the precursor liquid mixture being poured through the end of the tubular body in which the member 14 is normally fitted. After filling the chamber 12 with the requisite amount (e.g., about 2.5 ounces) of the precursor liquid mixture, the assembly of the member 14 and piston 26 can be inserted in position within chamber 12, the piston having been retracted on stem 24 to accommodate the poured volume of the precursor liquid mixture. Upon standing and cooling, the precursor liquid mixture gels or solidifies in situ to form the stick 20, which stick fits and is easily movable within chamber 12 as described above. Piston 27 can have one or more holes 29 provided in it to allow for escape of air when the assembly of member 14 and piston 26 is positioned as described. An inner integral ring 30 on the inner wall of tubular body 11 acts as a stop means to limit the retractable extent of piston 26.

A specific ectoparasite control composition (and stick) of the invention was made up of the components, and amounts thereof, set forth in Table 3.

Table 3

20	Component	Amount, g
25	Poly(isobutyl methacrylate-co-N-methyl-perfluorooctylsulfonamido-ethyl acrylate) -g- poly(dimethylsiloxane) substantive polymer, 25 wt% solution in Finsolv TN	33.4
30	C <sub>12-15</sub> -alkyl benzoate <sup>1</sup>	46.3
	Polyoxypropylene polyoxyethylene cetyl ether <sup>2</sup>	193.3
	Propylene glycol	193.3
	Deionized water	160.8
	Sodium stearate	32.2
	Pyrethrins insecticide concentrate <sup>3</sup>	26.7
	Piperonyl butoxide	114.2
35	Poly(dimethylsiloxane)cyclic tetramer <sup>4</sup>	33.3

<sup>1</sup>Finsolv TN solvent

<sup>2</sup>Procetyl AWS surfactant

<sup>3</sup>"Kenya Pyrethrum Extract Refined Concentrate"

40 <sup>4</sup>"VS-7207" silicone product of Union Carbide Corp.

The substantive polymer component listed first in Table 3 was prepared by polymerization of 10,000 molecular weight methacryloxypropyl terminated polydimethylsiloxane macromonomer (PDMS) with 2-(N-methyl perfluorooctylsulfonamido) ethyl acrylate (MeFOSEA) and isobutyl methacrylate (IBM). Briefly, 0.25 parts of 2,2'-azobisisobutyronitrile (AIBN) was added to a mixture of 25 parts of PDMS (Mw = 10,000), 5 parts of MeFOSEA, and 70 parts of IBM in 135 parts of ethyl acetate and 15 parts of isopropyl alcohol. This solution was purged with nitrogen and sealed in a bottle under nitrogen atmosphere. The bottle was heated in an Atlas Launder-O-Meter® (like a water bath) at 60°C for 24 hours. The reaction mixture prepared above (50 g) was added to Finsolv™ TN solvent (60 g) and both ethyl acetate and isopropanol were removed on a rotary evaporator to give 80 gram (25 wt% polymer) of a very viscous polymer solution in Finsolv™ TN solvent.

In preparing the formulation of Table 3, a mixture of the first five listed components was heated to 70°C while stirring the mixture in a 1000 ml beaker at 300-350 rpm. The sodium stearate was added to the mixture with stirring until it dissolved. The mixture was then cooled to 60°C while stirring. The pyrethrins insecticide concentrate, piperonyl butoxide, and silicone component were then added and the resulting final precursor liquid mixture was cooled to 55°C and poured into ten stick applicators (Item No. 9404 in the catalog, copyright 1988, of W. Braun Co.), the so-poured liquid solidified in stick form (each stick being about 2.5 oz. and about 3¼-inch long and ¾ inch wide) in each of the applicators upon standing at room temperature.

Other sticks of this invention were made as described above from formulations similar to that of Table 3 but using different anti-ectoparasite agents, namely, "Permanone 80" synthetic pyrethroid, "Dursban" organophosphate, "Sendran" carbamate, "DEET" repellent, and "Nylar" insect growth regulator.

The sticks prepared with the composition of Table 3 were clinically evaluated for effectiveness against fleas and ticks on infested dogs. The sticks were applied to the hair coat of the animals to provide 0.34 to 0.39 g/kg body weight. Against fleas, the sticks provide a 91% to 96% reduction of fleas through Day 13 post treatment when the sticks were both applied to the dogs "with" the lay of the hair (Method A) or "against" the lay of the hair (Method B). The sticks also provided 81% to 84% reduction of fleas through Day 16 post treatment. The sticks at 20 days post treatment provided a 78% reduction of fleas when the sticks were applied by Method A and a 58% reduction of fleas when the sticks were applied by Method B. Thus, the data show the ectoparasite control composition

of this invention was effective against fleas, the coating on the dogs having a residual effect over a prolonged period, which was attributed to the substantivity of the coating imparted by the acrylate polymer component. This effectiveness of the sticks was superior to a comparable stick formulation that omitted the acrylate polymer, in that the sticks of this invention extended the residual effectiveness by 7 days over the comparable sticks formulated without the substantive acrylate polymer.

In clinical tick evaluations, the sticks made from the formulation of Table 3 provided 85%, 67% and 29% reduction of ticks (R. sanguineus) through Days 6, 13, and 20, respectively, post treatment when the sticks were applied by Method A. When the sticks were applied by Method B, the reduction of ticks was 29%, 69% and 60% through Days 6, 16, and 20, respectively, post treatment. When compared again with the results obtained with sticks made from a comparable formulation that omitted the acrylate polymer, these data show in general that the sticks of this invention (Table 3 formulation) extended the residual effectiveness against R. sanguineus by 7 to 8 days (from 13 days to 20 days) over the comparable sticks formulated without the substantive acrylate polymer.

The sticks with the formulation of Table 3 were also clinically evaluated against adult D. variabilis on dogs and found to be effective for ectoparasite control, the data of such evaluation showing residual effectiveness at or near the 85% level through 6 days post treatment using Method B and superior in that respect to the results obtained with the comparable sticks whose formulation omitted the substantive acrylate polymer.

In carrying out the clinical evaluations described above, the animals remained healthy, did not appear stressed or uncomfortable, and showed no reaction atypical to the animal's behavior that could be deemed related to the presence of the stick coating.

Table 4 sets forth the formulation of three other ectoparasite control compositions of this invention that were prepared in stick form similarly to the sticks prepared from the formulation of Table 3.



Table 4

Components	Formulations		
	A	B	C
5 Copolymer of 35 wt % isooctyl acrylate, 45 wt % stearyl methacrylate, and 20 wt % acrylic acid, as a 30 wt% solution in Finsolv TN	2.34 g	1.99 g	1.82 g
C <sub>12-15</sub> alkyl benzoate (Finsolv TN)	3.90	4.30	4.95
Polyoxypropylene (5) polyoxyethylene (20) cetyl ether*	15.06	12.84	12.73
10 Propylene glycol	15.01	12.72	12.86
Deionized water	12.49	10.63	10.61
Sodium Stearate	2.50	2.00	2.00
15 Insecticide: "Kenya Pyrethrum Extract Refined Concentrate" "Permanone 80" synthetic pyrethroid "Dursban" chlorpyrifos, 99%	2.19	3.09	2.40
Piperonyl butoxide, 80%	9.08	-	-
Poly(dimethylsiloxane)cyclic tetramer**	0.69	0.62	0.68

20 \*\*Procetyl AWS" product of Croda Inc.

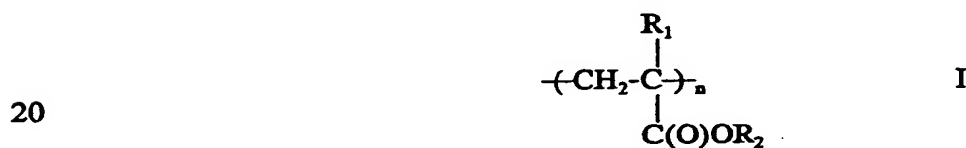
\*\*\*VS-7207" silicone product of Union Carbide Corp.

25 Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention.

## CLAIMS

1. An ectoparasite control composition comprising
- 1) an ectoparasite control agent,
  - 5 2) a water-insoluble acrylate polymer which imparts substantivity to the composition, and
  - 3) a gelling agent, wherein the composition is formed into a transferable solid stick adapted to be rubbed or spread by hand on the coat of a hair-coated animal to transfer or deposit a thin, solid, cosmetically acceptable, substantive coating of the ectoparasite control composition on the animal's coat to
  - 10 control ectoparasites which are on or come on the animal.

2. The composition of claim 1 wherein the ectoparasite control agent is a pyrethrin insecticide, the acrylate polymer is a polymer represented by the
- 15 general formula



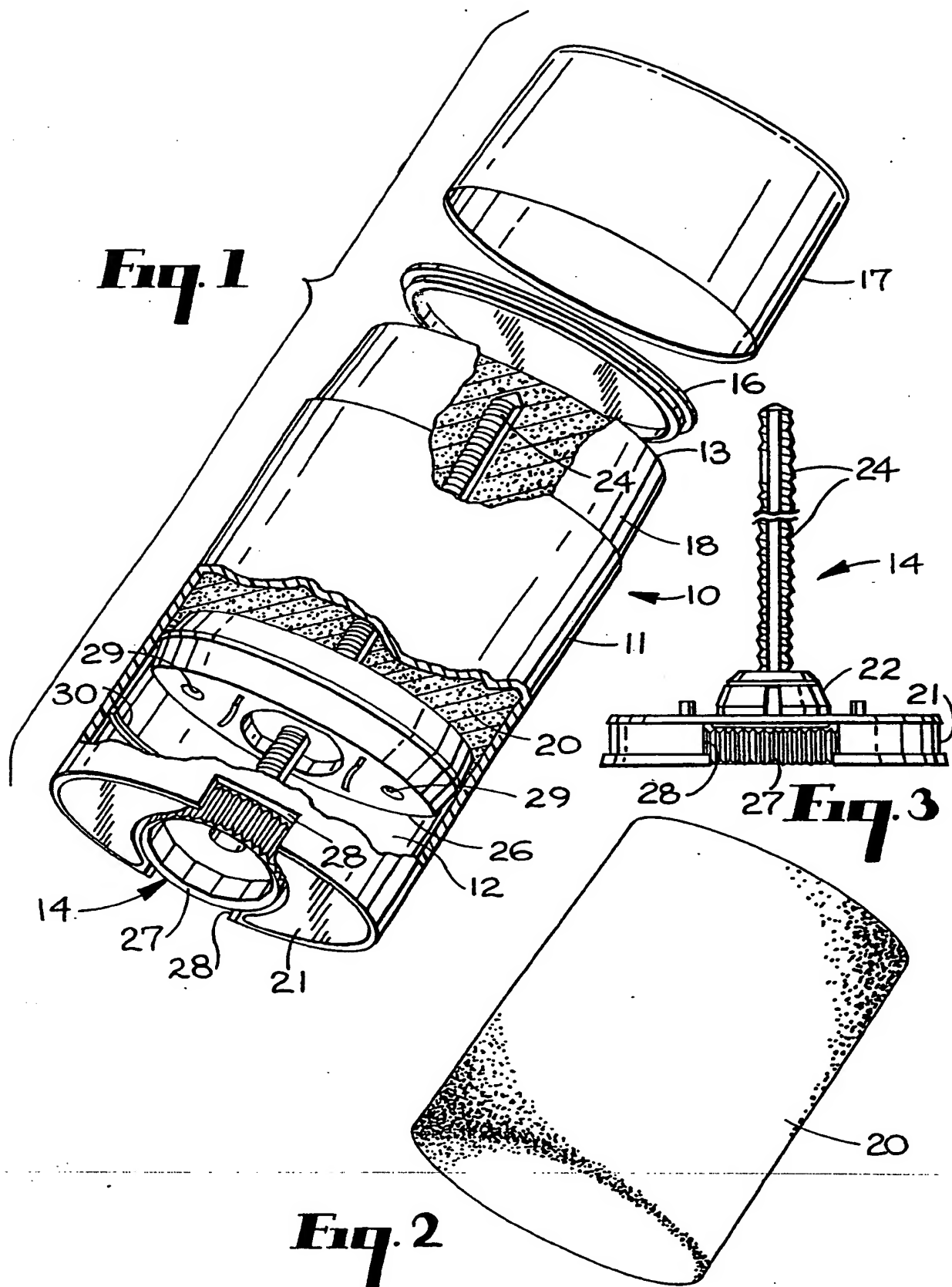
wherein:

- 25 n is an integer between 10 and 100,000 which represents the average number of repeating units, shown in the parenthesis, in the polymer;
- each R<sub>1</sub> in the repeating unit is independently a hydrogen atom or a methyl group; and
- each R<sub>2</sub> in the repeating unit is independently a hydrogen atom, a
- 30 hydrocarbon moiety, a fluoroaliphatic group, or a poly(dialkylsiloxane) group, and the gelling agent is sodium stearate.

3. The composition of claim 2, wherein the polymer is poly(isobutylmethacrylate-co-N-methyl-perfluorooctylsulfonamidoethyl
- 35 acrylate)-g-poly(dimethylsiloxane).

4. The composition of claim 1 further comprising water, an alcohol-free organic solvent and a surfactant.

5. The composition of claim 1 further comprising a lubricity agent.
6. The composition of claim 5 further comprising a silicone lubricity agent.
- 5 7. The composition of claim 2 further comprising a synergist for the pyrethrin insecticide.
8. The composition of claim 7 wherein the synergist is piperonyl  
10 butoxide.
9. A method of treating a hair-coated animal to control ectoparasites thereon comprising the step of applying an ectoparasite control composition of claim 1 on the coat of the animal.



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 94/13903

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 A01N25/24 A01N25/34

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CA,C,1 286 985 (MINNESOTA MINING AND MANUFACTURING COMPANY) 30 July 1991 cited in the application see claims ---	1-9
A	US,A,4 972 037 (J.E. GARBE ET AL.) 20 November 1990 cited in the application see column 4, line 51 - line 61 see column 12, line 4 - line 22 ---	1-9
A	EP,A,0 251 464 (BOOTS COMPANY) 7 January 1988 see claims ---	1-9
A	US,A,3 826 232 (T.E. DUFFY ET AL.) 30 July 1974 cited in the application -----	

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

21 March 1995

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		AU-A- 5989490	07-02-91
		CA-A- 2021580	08-02-91
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US-A-3826232	30-07-74	NONE	

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